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Improvement of fatigue resistance of a tool steel by surface treatments

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Abstract

30NiCrMoV12 quenched and tempered samples were coated with 4 µm of CrN produced by cathodic arc deposition technique. Micro-blasting treatment was performed on the coated samples, after the deposition, to remove defects like droplets from the surface. The fatigue limit was assessed and compared to that of the bare steel. A basic characterization of the coating/substrate was performed (hardness, roughness and adhesion). The residual stress distribution next to the surface of the samples was measured and its effect on the fatigue limit was evaluated. The micro-blasting treatment does not adversely affect the hardness and the adhesion of the coating nor the fatigue resistance of the coated samples.
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1. Introduction

CrN coatings have been extensively used as protective coatings on many types of tools and dies [1-5] thanks to their high hardness, excellent wear resistance and corrosion behaviour and good oxidation resistance with respect to several alloyed steels**.**

Studies on PVD coatings are often aimed at pointing out the effect of the thin coating on the wear and corrosion resistance.[6,7] However, it is also important to investigate the influence of the coating on the fatigue resistance of

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the base material when the coated component works under cyclic loading conditions.[8,9] Considering uncoated metallic materials, a mechanical surface modification is often obtained by shot peening.[10,11] This kind of treatment carried out with the proper applied pressure, could be interesting also in the case of PVD coated parts. Similar treatments, like micro-blasting, are already industrially applied to PVD coatings not with the aim of improving the fatigue behaviour but to increase the adhesion between the metallic substrate and the coating or to enhance the wear resistance of the tools. It is well known that a treatment of micro-blasting carried out on carbide cutting tools before the coating deposition increases the hardness and the compressive residual stress at the surface and, as a consequence, the in-service life of the component.[12] Bouzaky et al. stated that, with the proper process parameters, a micro-blasting treatment after coating deposition can improve the tool life, even in the case of tools reconditioned several times.[13,14] On the other hand, if wrong working conditions are chosen, the treatment can lead to delamination of coating resulting in drastic reduction of the strength of the tool.[15]

In this work, the fatigue resistance of a tool steel coated with a CrN monolayer coating was studied. The coating was fully characterized in terms of roughness, defect distribution, hardness, adhesion and residual stress distribution. Concerning the fatigue characterization, data has been collected for the bulk material as well as for the coated parts in order to evaluate the effectiveness of a micro-blasting treatment on the coating surface.

1. Materials and Methods

Chemical composition and mechanical properties of the tool steel considered in this work are shown in Table 1 and Table 2 respectively.

Table 1. Chemical composition (wt%) of the considered steel

C	S	P	Mn	Cr	Ni	Mo	Cu	Si	Al	Fe
0.28	0.002	0.008	0.62	0.85	3.06	0.51	0.17	0.31	0.023	Balanced

Table 2. Mechanical properties of the considered steel, *Transversal, **Longitudinal

YS [MPa]	UTS [MPa]	YS/ UTS	Elongation %	Red. of Area %	Impact test		Hardness HRC
					KV T* [J]	KV L** [J]	
1192	1376	0.87	12.4	50.15	25	25	42

The steel was supplied in a quenched and tempered condition fixed by the request of a minimum hardness of 42 HRC to assure a proper wear resistance even in the case of the injection molding production where it is often used. This steel is characterized by a low percentage of Carbon to guarantee a good weldability and a quite a high presence of Nickel to improve the quenchability and the toughness of the final component.

Fatigue specimens were machined according to ISO1143-2010 standard. Three sets of samples were then considered:

- Bare steel;
- Bare steel coated with CrN (CAE1);
- Bare steel coated with CrN + micro blasting treatment (CAE1MB).

CrN coating was deposited with Cathodic Arc Evaporation (CAE) technique with the thickness of 4±0.2 µm. For CAE1MB configuration, a micro-blasting treatment after the deposition of the coating was made. This treatment is commonly used by industries in the case of the Cathodic Arc Evaporation technique. In fact, coating deposited by this technique are characterized by a high number of surface defects [16] that could worsen the adhesion between the layers in the multi-layered coating. A micro-blasting after the deposition of each layer results effective in removing droplets from the coating surface and, therefore, it improves the adhesion between the layers.[17] The micro-blasting also results in a significant increasing in the wear resistance of coated samples tested by pin on disk apparatus.[18]

In the present paper, the fatigue resistance was investigated as well as the effect of a final micro-blasting on the coated surface.

The micro-blasting treatment was performed with silica media of a size between 70 and 100 µm and an average hardness of 47 HRC. The impact angle was 90° and the pressure was set to 0.2 MPa. Further details of the micro-blasting treatment can be found in [7].

On all sets of samples both roughness and residual stress measurements were carried out. On coated samples, nanoindentation and scratch tests were also executed to determine the mechanical properties and evaluate the adhesion of the coating to the substrate.

Roughness measurements were carried out with a stylus profilometer having a tip radius of 5 µm. Residual stress field in the bulk material was measured using a XRD Enixè TNX diffractometer according to ASTM E2860-12 and UNI EN 15305 – 2008 standards.

Young's Modulus and coating hardness were measured by a CSM Table Top Nanoindentation Tester (TTX-NHT) equipped with diamond Berkovich indenter. An applied load of 50 mN and a load rate of 100 mN/min were used.

Scratch tests were carried out using a CSM Revetest Xpress with a Rockwell indenter having a tip radius of 200 µm. A scratch length of 10 mm, a loading rate of 1 N/s and a maximum applied load of 100 N were imposed. In particular, the adhesion was defined in terms of critical loads (LC). LC1 corresponds to the load needed to cause the first crack in the coating while LC2 is the load linked to the full coating delamination.

All the groups of samples were tested by rotating bending fatigue tests following the Stair Case Method defined in UNI 3964/85 standard and the influence of coating presence and micro-blasting treatment on the fatigue limit of the bare steel was evaluated.

Both fracture surfaces and metallographic samples were observed through a LEO EVO 40 scanning electron microscope (SEM) equipped with EDS probe. The distribution and the chemical composition of defects present on the coated surface were assessed and the nucleation points of fatigue fracture were studied.

2. Results

2.1. Roughness measurements

Table 3 summarizes the Ra and Rz results. In order to obtain a statistical dispersion of the results each data is the average of at least 10 measurements obtained on a minimum of two samples for each examined set.

Table 3. Roughness measurements results

	Ra [µm]	Rz [µm]
Bare steel	0.05 ± 0.02	0.42 ± 0.13
CAE1	0.05 ± 0.01	0.47 ± 0.06
CAE1MB	0.07 ± 0.01	0.55 ± 0.03

The deposition of a coating by CAE technique does not affect the roughness of the substrate whereas micro-blasting treatment makes it a little worse both in terms of Ra and Rz.

2.2. Residual stress measurements

The results of the experimental residual stress measurements are reported in Table 4. All examined conditions show a not negligible compression at the interface with the substrate. The presence of a compression state in the substrate before the deposition has to be associated to the production process of the samples subjected to quenching and tempering, and then to cutting operations until the final polishing step. Being the strong compression in the

CrN-PVD coating (from 2 GPa to 6 GPa) a typical feature of PVD technique, [19,21] and since it was found that the micro-blasting treatment does not change the stress state of the coating,[22] particular attention was devoted to the stress field characterizing the substrate. It was found that the CAE technique leaves almost unaltered the initial value of compression of the bare steel and that the treatment of micro-blasting, although it is performed only on the coating and with an applied pressure that does not cause cracking in the coating, increases the compression in the substrate.

Table 4. Residual stress results

	Substrate [MPa]
Bare steel	-511 ± 28
CAE1	-482 ± 32
CAE1MB	-799 ± 37

2.3. Nanoindentation testing results

The results of the nanoindentation testing are summarized in Table 5.
The CrN coating deposed using the CAE technique leads to a stiffness of about 300 GPa and a hardness of about 2000 HV. These data remain stable even after the micro-blasting treatment due to the low micro-blasting pressure which is not able to induce a significant effect on the CrN coating.[7, 17]

Table 5 : Nanoindentation results

	Stiffness [GPa]	Hardness [HV]
CAE1	293 ± 8	2010 ± 95
CAE1MB	305 ± 36	2004 ± 110

2.4. Scratch tests results

In Table 6 a comparison between the load that induces the first damage in the coating (LC1) and the one causing the total detachment of the CrN coating are presented. The micro-blasting treatment does not affect the obtained results, contrarily to the expectation. In fact, the main use of micro-blasting in the industrial production of thin PVD coatings is aimed at increasing the adhesion between the substrate and the coating.

Table 6 : Scratch test results

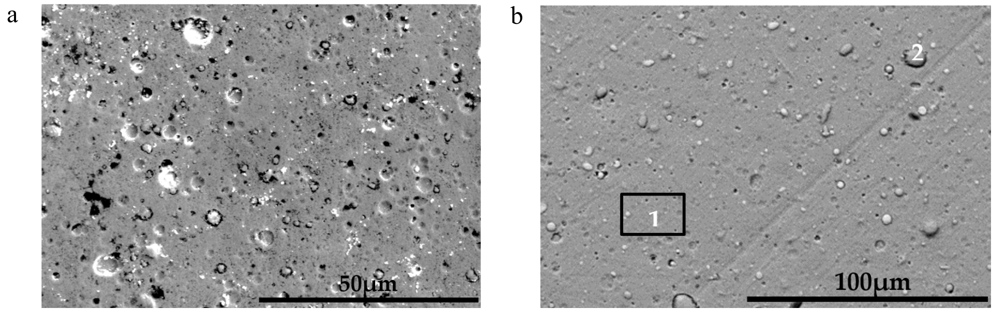
Critical load [N]	LC1	LC2
CAE1	14.9 ± 1.5	50.6 ± 1.3
CAE1MB	14.0 ± 1.5	49.9 ± 0.9

2.5. SEM analyses

Figure 1 shows the surfaces of the coated samples before and after the micro-blasting treatment.

The surface of the coated samples (both as deposed and after micro-blasting) shows a quite homogeneous distribution of chromium metallic droplets and several hemispherical cavities of size ranging from one micrometer to tens micrometers. The observation of the sample surfaces after micro-blasting, presents a lower amount of droplets than the as-deposited condition. Consequently, the surface is characterized by the presence of cavities due to the removal of large droplets emerging from the surface. Consequently, a slightly increase in roughness can be reported, especially in terms of Rz.

The cross section of the samples (Fig. 2) shows the absence of micro voids or precipitated particles in the coating thickness.



Spectrum	N	Cr
1	12.8	87.2
2		100.0

Fig. 1 Coated surfaces: a) CAE1MB; b) CAE1, EDS analysis (All results in weight %)

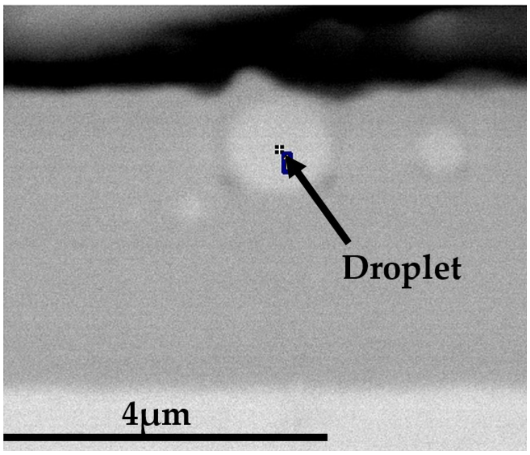


Fig. 2. Section of CAE1: example of a droplet located next to the surface.

2.6. Fatigue tests results

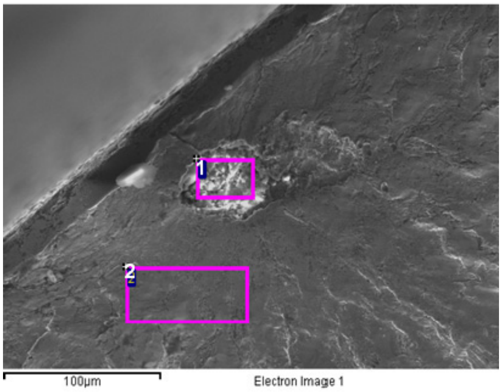
Rotating bending fatigue tests were carried out in air, at room temperature on the bare steel material, as well as on coated samples with and without micro-blasting treatment. The fatigue tests were set to run up to 2×10^6 cycles; in some cases, the fracture occurred before the end of the test. Fatigue limits obtained from the tested samples are summarized in (Table 7).

As already found in the literature [22, 23] , the presence of a PVD coating increases the fatigue limit of the base material mainly for the presence of a high level of residual compressive stress affecting the coatings, that progressively reduces its value to a low tensile stress at the interface with the bulk steel.

In the present work it was also found that a micro-blasting treatment increases the fatigue limit by a further 6% with respect to the as-deposed coating and reduces of an order of magnitude the results dispersion. The fracture surfaces were also analyzed by means of SEM in order to better investigate the nucleation point. For all the fractured specimens the fatigue crack nucleated at a large nonmetallic inclusion or at a cluster of inclusions located inside the steel near the coating/substrate interface (Figs. 3,4).

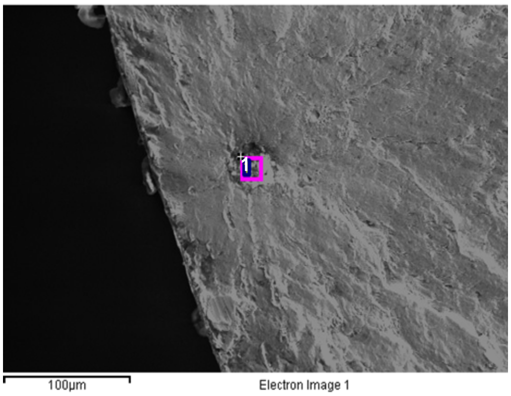
Table 7 : Fatigue tests results

	Bare steel	CAE1	CAE1MB
Fatigue Limit [MPa]	491 ± 18	645 ± 11	683 ± 3



Spectrum	O	Mg	Al	Si	S	Ca	Cr	Mn	Fe	Ni
1	44.51	5.56	37.48	0.71	0.38	9.18		0.61	1.57	
2							0.75	0.64	95.53	3.08

Fig. 3 Fracture surfaces: CAE1, P = 660 MPa, EDS analysis; (All results in weight %)



Spectrum	O	Mg	Al	Ca	Cr	Fe
1	38.70	2.44	37.92	13.48	0.93	6.53

Fig. 4 Fracture surfaces: CAE1MB, P = 680 MPa, EDS analysis; (All results in weight %).

The inclusions found were mainly mixed oxides of calcium, aluminum and magnesium. Comparing non-metallic inclusions similar for chemical composition and not too different in shape and size, some considerations can be done for coated samples regarding the position of the fatigue nucleation cracks.
Figure 3 reports, as an example, the specimen without treatment of micro-blasting where a crack nucleated from an inclusion located at 63 μm from the interface for an applied stress of 660 MPa. In figure 4 the specimen subjected to micro-blasting treatment can be observed, characterized by a crack starting from an inclusion located at 84 μm from the interface for an applied stress of 680 MPa.

These results can be interpreted considering the gradient of the residual stresses that, starting at values that for a CrN-PVD coated sample can reach 3 GPa, resulted considerably reduced due to the coating/substrate interface up to few hundreds of MPa just below the interface. Despite the measured increase of the compressive stresses at the substrate/coating interface caused by the treatment of micro-blasting was not negligible (about 300GPa of compression), this treatment is not able to significantly increase the depth of the zone where the residual stress data change from compressive to tensile condition and, for such a reason, a limited increase in the fatigue limit was measured.

3. Conclusions

In the present paper, the fatigue behaviour of a tool steel was considered, and the effect of a PVD CrN coating on the enhancing of the fatigue limit was studied. In addition, the effect of a micro-blasting treatment carried out after the coating deposition was investigated. In fact, it is known that such a process is effective in removing defect such as droplets from the coating surface but no data have been published on the fatigue behaviour of PVD coated parts undergoing micro-blasting.

The experimental results showed that the micro-blasting treatment only removes surface defects like large droplets and does not influence either the mechanical properties of the coating or the adhesion between the coating and the substrate. On the contrary, slightly increased roughness values in terms of both Ra and Rz were detected. This condition does not affect the fatigue resistance for the PVD coated tested specimens because of the high compressive residual stress value, due to the coating deposition, that always assures nucleation of the fatigue fracture in the steel next to the interface with the coating but surely not in the coating. Yet, the condition can be considered dangerous because the body behavies as stress concentration points were present.

The obtained results allow to state that the micro-blasting treatment can be used for those components that must guarantee a good resistance to cyclic loads besides a high wear resistance.

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